

**Georgia Department of Natural Resources**  
**Environmental Protection Division Laboratory**

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SOP 2-004 Rev. 3  
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**SW846-3010A: Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis  
by ICP Spectroscopy**

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1 Scope and Application

Method SW846-3010A is used for the preparation of aqueous samples, EP and TCLP extracts, and wastes for analysis by ICP. Samples prepared by this procedure are analyzed by SW846-6010B or SW846-6020

<u>Compound</u>	<u>CAS No.</u>
Aluminum	7429-90-5
Antimony	7440-36-0
Arsenic	7440-38-2
Barium	7440-39-3
Beryllium	7440-41-7
Cadmium	7440-43-9
Calcium	7440-70-2
Chromium	7440-47-3
Cobalt	7440-48-4
Copper	7440-58-8
Iron	7439-89-6
Lead	7439-92-1
Magnesium	7439-95-4
Manganese	7439-96-5
Molybdenum	7439-98-7
Nickel	7440-02-0
Potassium	7440-09-7
Selenium	7782-49-2
Silver	7440-22-4
Sodium	7440-23-5
Strontium	7440-24-6
Thallium	7440-28-0
Tin	7440-31-5

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Vanadium	7440-62-2
Zinc	7440-66-6

**1.2 Restricted Procedure**

This procedure is restricted to use by an analyst experienced in the handling of hazardous materials. Additionally, the analyst must complete the requirements of the GAEPD Initial Demonstration of Analyst Proficiency prior to the analysis of actual samples. Analysts are further warned that performance of this analysis involves the use of potentially hazardous chemicals; refer to the GAEPD Chemical Hygiene Plan for additional information regarding chemicals required by this method.

**2 Definitions**

Refer to Chapter 3 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions.

**3 Interferences**

Interferences are discussed in the analytical method.

**4 Safety**

Refer to Laboratory Chemical Hygiene Plan, online revision

**5 Apparatus and Equipment**

- 5.1 Virgin 50 ml flat bottom HDPE graduated tubes with screw caps for digesting samples.
- 5.2 Various size pipetters capable of delivering volumes ranging from 1.0 to 5000  $\mu$ L and an assortment of high quality pipet tips.
- 5.3 Hot block capable of maintaining a temperature of 95°C.
- 5.4 Balance capable of measuring to 0.1 mg.
- 5.5 Steel cabinet centrifuge with electric timer and brake.
- 5.6 HDPE watch glasses.
- 5.7 Plastic wash bottles.

**6 Reagents and Standards**

- 6.1 Concentrated nitric acid (sp. gr. 1.41), reagent grade.
- 6.2 1:1 Hydrochloric acid, reagent grade.
- 6.3 18 M $\Omega$  water.
- 6.4 Spiking solution for preparing all matrix spikes and laboratory control samples.

**7 Sample Collection**

Water samples and liquid waste samples for metal analysis are collected in 500 ml narrow mouth plastic (HDPE) bottles. Samples are preserved with sufficient HNO<sub>3</sub> to lower the pH below 2. One to two bottles are required for each sample.

**8 Calibration**

Not applicable.

**9 Quality Control**

Refer to analytical method

**10 Procedure**

- 10.1 Transfer 50mL aliquot of well mixed , acid preserved sample to a 50ml Hotblock digestion tube. (When necessary, smaller sample aliquot volumes may be used)
- 10.2 Add 1.5 mL concentrated nitric acid to the measured sample.
- 10.3 Transfer 50 mls of 18 MΩ water each for the matrix blank, laboratory control sample (LCS), laboratory control sample duplicate (LCSD) to 50 ml Hotblock digestion tubes.
- 10.4 Transfer an additional 50 mL of one routine well mixed sample for the matrix spike (MS) and another 50 mL for the matrix spike duplicate (MSD) for every ten samples digested in the batch.
- 10.5 Place digestion vessels in the hotblock (located in a fume hood) and cover with a ribbed watch glass. Adjust the temperature to approximately but no higher than 95°C. Place a thermometer in a digestion vessel filled with 50mL water to monitor the temperature. If a sample boils or sample is lost due to spattering discard the sample and redigest
- 10.6 Reduce the volume of sample aliquot to about 5 mL by gentle heating at 95°C. DO NOT BOIL.
- 10.7 Remove the samples from the hotblock and cool. Uncover the samples and add another 1.5 mL concentrated nitric acid to each. Return the samples to the Hotblock and cover with a non-ribbed watch glass.
- 10.8 Continue heating, adding additional nitric acid as necessary, until the digestion is completed (the digestate is light in color or does not change in appearance with continued refluxing). Any sample that dries out must be redigested.
- 10.9 Uncover the samples and evaporate to about 5 mL.
- 10.10 Remove the samples from the Hotblock, cool, and add 5 mL 1:1 hydrochloric acid to each sample. Cover the samples with a watch glass, place back in the Hotblock, and reflux for 15 minutes.
- 10.1.11 Bring the sample volume up to 50 mls with 18 MΩ water and mix well.
- 10.1.12 Allow any undissolved material to settle overnight, or centrifuge a portion of the prepared sample until clear. (if the sample still contains suspended material that would clog the nebulizer, a portion of the sample may be filtered prior to analysis.)

**11 Evaluation of the Linearity of the Initial Calibration**

Not applicable.

**12 References**

- 12.1 *Test Methods for Evaluating Solid Waste, SW846*, USEPA Office of Solid Waste, revision 4, 1996.

- 13      Practical Quantitation Limits (PQLs), Precision and Accuracy Criteria, and Quality Control Approach**  
Not applicable

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